Rec'd PCT/PTO 05 JAN 2005
PCT/IB03/02980

02.07.03

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Patentanmeldung Nr. | Patent application No. | Demande de brevet n°

02077784.3

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Anmeldung Nr.:
Application no.:
Demande n°:
02077784.3

Anmeldetag:
Date of filing:
Date de dépôt:
10/07/02

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Bezeichnung der Erfindung:
Title of the invention:
Titre de l'invention:
Transparent polycrystalline aluminium oxide

In Anspruch genommene Prioriät(en) / Priority(ies) claimed / Priorité(s) revendiquée(s)

Staat:
State:
Pays:

Tag:
Date:
Date:

Aktenzeichen:
File no.
Numéro de dépôt:

Internationale Patentklassifikation:
International Patent classification:
Classification internationale des brevets:
/

Am Anmeldetag benannte Vertragstaaten:
Contracting states designated at date of filing:
Etats contractants désignés lors du depôt:
AT/BG/BE/CH/CY/CZ/DE/DK/EE/ES/FI/FR/GB/GR/IE/IT/LI/LU/MC/NL/

Bemerkungen:
Remarks:
Remarques:

Transparent polycrystalline aluminium oxide

The invention relates to technical ceramics and to highly dense translucent and transparent aluminium oxide structures for applications where, e.g. in the lighting industry, a fine crystal size has to be obtained and stabilised for use at temperatures of 800 °C or more. The invention is also related to an electric lamp having a discharge tube with a wall of such ceramic.

Sintered transparent alumina ceramics consisting of chemically and thermodynamically stable corundum phase (α-Al_2O_3) have been available for several decades, Traditionally, they are produced starting from very fine-grained transitional alumina raw powders and obtain a high sintering density by annealing at very high temperatures > 1600 °C. As a result, the ceramic microstructures are coarse with crystal sizes typically > 15 µm. As a consequence of this coarse microstructure, these materials exhibit even in thin components only translucency but no transparency. Besides the known ceramics have a relative low bending strength, usually less than 300 MPa.

Transparency of a ceramic component is herein defined as having a value for real in-line transmission RIT of at least 30%, wherein the real in-line transmission RIT is measured over an angular aperture of at most 0.5^0 at a sample thickness of 0.8 mm and with a monochromatic wave length of light λ.

In literature it is common practise for optical properties to use total forward transmission (TFT) and in-line transmission (IT), the latter measured with commercially available spectrometers. As these have angular apertures of several degrees the so measured IT includes a large amount of multiple forward scattered light. As a consequence, for scattering samples, both TFT and IT will always result in values much higher than the value of RIT for the same sample. Due to the multiple reflective character of the light measured with TFT and IT it is not possible to establish any quantitative relationship with the RIT. It is however possible to compare values of real in-line transmission which have been taken at samples with a thickness, which differs from the 0.8 mm as defined. For a sample 1 with

thickness d_1 and RIT value T_1 and a second sample having a thickness d_2, the value T_2 of the RIT satisfies the relation

$$T_2 = (1-R) * [T_1 / (1-R)]^{d_2/d_1} \qquad (1)$$

with R the coefficient of surface reflection which is for alumina 0.14 (incorporating the reflection on *both* surfaces). Due to reflection losses a transmission value, either RIT, TFT or IT cannot exceed a value of 86%.

The inventors have established that for a ceramic sample having a very small porosity as well as small pores, at least smaller than 0.01 % respectively <100nm the real in-line transmission RIT is correlated to the samples structure. When measured according to the above stated definition the obtained RIT fulfils the expression

$$RIT = (1-R)\exp\left(-\frac{3\pi^2 G d \Delta n^2}{2\lambda_0^2}\right), \qquad (2)$$

with R the coefficient of surface reflection (for Al_2O_3 0.14), d the sample thickness, G the average crystal size, Δn the effective birefringence of alpha-alumina (0.005) calculated as the weighted average of the refractive index differences between each of the main optical axes and λ_0 the wavelength of the monochromatic incident light in vacuum. With higher porosity percentages and larger pore sizes, the measured RIT results in significant-smaller values than predicted by the above expression.

To overcome a low bending strength with known ceramics, an early Japanese investigation applied a slip casting approach in combination with pressureless pre-sintering and hot-isostatic post-densification (HIP) to obtain high-strength and translucent sintered products with fine crystal sizes of 2-5 μm (H. Mizuta et al., J. Am. Ceram. Soc. 75(1992)2, 469-473). No RIT was measured but the maximum IT was 46 % (at 1mm sample thickness, visible to infrared light - no wave-length given) observed at an average crystal size of 5 μm and was associated with a 3-point bending strength of 786 MPa.

Only slight improvements were observed when the crystal sizes are reduced towards the sub-micrometer range as described by K. Hayashi et al. (Mater. Trans., JIM, 32(1991)11, 1024-1029). Dense samples produced with an average crystal size of 0.82 μm by injection moulding, pre-sintering and HIP exhibited a strength of 812 MPa and an IT (at 500 nm wave length) of 78 % recorded at a sample thickness of 0.5 mm thickness.

The purity of the alumina in these Japanese investigations was 99.99 %. The HIP process in these investigations is done at a temperature of about 1250 to 1280 ^{0}C giving rise to an additional difficulty, however, when the ceramics are intended for use in a

discharge lamp, a discharge tube of such discharge lamp is operated at temperatures ranging from 1100 to 1300 °C. Any technical use of these sintered products at temperatures similarly high or even higher than the HIP temperatures will unavoidably coarsen the described highly pure alumina microstructures. Whereas several additives like for instance MgO and ZrO_2 have been reported to retard crystal growth in annealing alumina ceramics, the precise effects are often unclear.

According to EP 1053983 A2 related to translucent polycrystalline ceramics with mean facet lengths no longer than a maximum wave length λ of the light (which for λ = 600 nm e.g. means a crystal size of about 0.6 μm since the facet length is about half of the average crystal size), an additive of only 0.05 mol-% ZrO_2 as a sinter dopant in transparent sintered alumina ceramics has a degenerating effect on both the optical transmittance and the strength and the hardness compared with samples without ZrO_2. For 0.5 mm thin discs and λ < 800 nm, the value measured for what is called linear transmission, which in this case can be compared to the real in-line transmission RIT, decreases to 25 % compared with 40 % measured for a zirconia free microstructure with usual MgO dopant (0.1 mol-%). A RIT value of 25 % for a thickness d of 0.5 mm corresponds according to the relation (1) to a value of 12 % for thickness d=0.8 mm. For a zirconia free microstructure the corresponding value for a thickness d=0.8 mm is 25 %.

A transparent Al_2O_3 component with a value for the RIT measured over an angular aperture of at most 0.5^0 at a sample thickness of 0.8 mm and with a monochromatic wave length of light λ of at least 30 % having an acceptable strength is therefore unknown. That is a problem. It is therefore the objective of the present invention to dissolve the problem and to provide a component, which overcomes the previously mentioned limitations.

The present invention provides high-strength translucent and transparent polycrystalline alumina components which associate a zirconia (ZrO_2) additive to stabilise a fine crystal size ≤ 2 μm or, preferentially, ≤ 1 μm for use at temperatures of 800 °C or more with a high real in-line transmission > 30 %, preferentially > 40 % and more preferentially > 50 %, measured at a sample thickness of 0.8 mm, over an angular aperture of at most 0.5^0 and with a monochromatic wave length of light λ, preferably of 645 nm.

The result that a value of RIT > 30 % is obtained in spite of the presence of zirconia additives in concentrations large enough to prevent crystal growth on high

temperature annealing is surprising and clearly is in disagreement with the previous state of the art. It is enabled here by the combination of very small crystal sizes with an extremely high relative density > 99.95 % implying a very small residual porosity.

Disclosed are polycrystalline alumina components with at least initially an average crystal size < 2 μm, a purity > 99.5 % Al_2O_3 and optionally containing up to 0.3 wt.-% of MgO characterised by an additive of at least 0.001 wt-% ZrO_2 and by a relative density which at these ZrO_2 concentration is > 99.95 % with a real in-line transmission RIT > 30 % measured at a sample thickness of 0.8 mm over an angular aperture of at most 0.5^0 and with a monochromatic wave length of light λ, preferably of 645 nm.

Disclosed is also a process of manufacturing such new alumina ceramic components based on colloidal dispersion providing an extremely high degree of dispersion homogeneity obtained by the application of dispersing methods, selected from the group of stirring, milling, and ultrasonification, and introducing at least 0.001 wt-% ZrO_2 additive either as a solution of a zirconium salt, as a finely dispersed ZrO_2 powder, or by wear from ZrO_2 containers, milling discs or beads followed by a hot-isostatic pressing (HIP) as a post-densification treatment.

It is believed that on sintering, the ZrO_2 dopant induces two opposite effects on the microstructural development of the transparent alumina: as first effect grain boundary pinning reduces crystal growth of the Al_2O_3 crystals and may, by this means, increase the RIT characteristics of the sintered products; this effect prevails at lower concentrations and fine ZrO_2 crystal sizes < 100 nm. On the other hand as second effect, ZrO_2 increases the temperature required for densification of Al_2O_3, and this higher temperature might be associated with crystal growth and decreasing RIT data. The inventors have, however, observed that increasing the ZrO_2 concentration up from 0.001 to about 0.5 wt.-% will result in an increased value for the RIT of the thus formed alumina components to values as high as 69 % despite an increase of the sintering temperature by 100-200 °C (with a constant temperature of the subsequent HIP treatment of 1200 °C). For a dopant concentration of less than 0.001wt-% there was no practicable effect resulting in continuation of crystal growth at temperatures of 800 °C and above. Dopant concentrations above 0.3 wt-% up to 0.5 wt-% result in a further increasing sintering temperature and thus coarsening of the microstructure and thus a lowering effect on the RIT but still result in values for the RIT above 30 %.

However in this range an increase in the zirconia concentration goes hand in hand with a decrease of the RIT.

With concentrations > 0.5 wt-% ZrO_2 result in decreased values for the RIT below 30 %.

Preferably in an embodiment of the invention the ZrO_2 additive has a concentration between 0.1 wt-% to 0.3 wt-%, boundaries inclusive.

A preferential embodiment of the invention is polycrystalline alumina components with initially an average crystal size < 1 μm and a real in-line transmission RIT > 40 % measured at a sample thickness of 0.8 mm measured over an angular aperture of at most 0.5^0 and with a wave length of light λ, preferable of 645 nm.

With their extremely homogeneous microstructure and their small sub-μm crystal size at a high relative density > 99.95 %, the alumina components exhibit outstanding mechanical properties. Their macrohardness HV10 (measured with a Vickers pyramid at a testing load of 10 kgf) is at least 19.5 GPa, and their 4-point bending strength is 550 MPa or more.

EXAMPLES

The invention is further elucidated by examples with reference to a drawing which shows a discharge tube with a ceramic wall.

Example 1

An aqueous slurry with a solid loading of 41 wt.-% was prepared at pH = 4 from TM-DAR corundum powder [average particle size 0.2 μm; make Boehringer Ingelheim Chemicals, Japan] without any further additives. A high degree of dispersion was obtained after at least 1 day of ultra sound or at least half a day of wet ball milling, using milling beads that could not give rise to contaminations other than alumina or oxydizable wear. The dope ZrO_2 was then introduced by the addition of pure and finely grained ZrO_2 powder with a solid loading of 20 %, dispersed in water and stabilised by nitrate ions. The average particle size of the dope is preferably chosen smaller than the alumina crystal size obtained after sintering and HIP treatment. In the described example it was chosen 100nm. Reference samples without ZrO_2 were prepared in the same way, except that no dope was added.

The thus obtained suspensions were without further degassing either pressure cast at a pressure of 4 bars using a Millipore hydrophilic membrane with an average pore diameter of 50 nm, or slip cast on a porous mould with an average porosity of about 50 %

and an average pore size of about 100 nm. After consolidation the pellets were dried in air for about 4 hours and hereafter further dried in a stove at a temperature of 80 °C for 4 more than hours. The dried compacts were calcined at 600 °C for 2 hours in pure oxygen to remove pollutants. Hereafter the pallets were sintered at a sinter temperature (T_s) ranging from 1150 °C to 1350 °C in either oxygen, vacuum or humidified hydrogen (dew point 0 °C). Pellets with a density higher than 96 % were given a subsequently HIP treatment at a temperature of 1200 °C at a pressure of 200 Mpa for at least 2 hours. The pellets were ground on both parallel sides, first with successively finer diamond grains of finally 3 μm. The final thickness of the discs was 0.8 mm.

The real in-line transmission (RIT) of the samples was measured using a red diode laser with a wavelength λ of 645 nm and a detector at a distance from the illuminated sample of at least 1 meter to ensure an angular aperture of 0.5°. Also the total forward transmission (TFT) was measured. The results are shown in Table I.

TABLE I

Sample no	ZrO_2 Wt-%	T_s (°C)	Density after sintering (%)	HIP-time (h)	RIT (%)	TFT (%)
1	0	1200	97.7	2	46.7	77.6
2	0	1250	99.7	2	45.1	74.9
3	0.01	1250	97.4	2	56.3	75.5
4	0.01	1300	99.6	2	49.9	73.4
5	0.04	1300	98.0	2	60	75.4
6	0.04	1350	99.9	2	47.1	72.8
7	0.1	1320	96.5	2	61.4	75.2
8	0.1	1350	99.7	2	59.2	76.1
9	0.3	1300	97.8	24	69.2	76.8
10	0.3	1350	100	24	55.6	73.2
11	0.5	1350	98.5	24	47.9	65.4
12	0.5	1400	99.3	24	20.9	48.8
13	1.0	1350	98.4	24	15.5	42.9
14	1.0	1400	98.7	24	3.5	29.4

The samples no 1,3,5,7,9,11and 13 were sintered at a lower range of possible sinter temperatures with which however it turned out still to arrive at a minimum density before the HIP treatment of 96 %. Comparison of these samples shows that up to 0.3 wt-% zirconia oxide the RIT value increases with increasing zirconia concentration, opposite to the state of the art. Apparently the resulting crystal size becomes smaller with increasing dopant, notwithstanding an increase in required sinter temperature. On the other hand the results in Table I show that for an equal amount of dopant an increase of the sinter temperature will in general result in a lower value for the RIT. In particular, this is significant in the case of a low dopant content and with dopant concentrations above 0.3 wt-%. Comparison the results of the samples 11 and 12 teaches that at the upper limit of the zirconia oxide concentration of 0.5 wt-%, an slight increase of the sinter temperature results in an dramatic drop in the resulting value for the RIT to below 30 %.

From the results of the samples 13 and 14 it is evident that increase of the zirconia oxide dopant above 0.5 wt-% will not result in a ceramic body with the properties accordinr to the invention.

As high intensity discharge lamps like sodium and metal halide lamps are operated at temperatures ranging between 1100 °C and about 1300 °C, the influence of an increased temperature on the crystal size is of importance. Further samples have therefor been treated at increased temperatures for some time after which the average crystal size was measured again. The result is shown in Table II.

TABLE II

	Average crystal size (µm)	
Annealing temperature for 24 hours (°C)	0 wt % ZrO_2	0.1 wt % ZrO_2
1225	-	0.5
1250	1.09	-
1275	1,49	-
1300	1.89	-
1350	-	0.93
1400	-	1.82

The Table II shows that a ZrO_2 concentration significantly retards the increase of the average crystal size and thus has a favourable influence on maintaining a high value for the RIT.

In another simulation the longer term influence of increased temperature on the crystal size has been investigated. The simulation is based on the model as disclosed in J. Am. Ceram. Soc. 73(1990) 11, 3292-3301. The effect on a sample having a ZrO_2 dope of 0.1 wt % is shown in Table III.

TABLE III

	Average crystal size(µm)			
Temperature(°C)	24 hours	100 hours	1000 hours	10,000 hours
1100	0.55	0.55	0.56	0.64
1150	0.55	0.56	0.62	0.97
1200	0.56	0.60	0.87	1.74
1250	0.62	0.78	1.48	3.14

The results in Table III clearly show that the enduring effect from the ZrO_2 on retarding of crystal growth under temperature conditions prevailing in operative lamps results in times of the sample having average crystal sizes < 2 µm being comparable to common lamp lifetimes.

Example 2

An aqueous slurry with a solid loading of 75 wt.-% and a monomer content of 3.5 % (acrylamide [AM] with n,n'-methylene-bis-acrylamide [MBAM] at a weight ratio AM:MBAM of 24:1) was prepared at pH = 4 (adjusted with HNO_3) from TM-DAR corundum powder [average particle size 0.2 µm; make Boehringer Ingelheim Chemicals, Japan] with an additive of 0.03 wt.-% MgO (introduced as an equivalent amount commercial spinel Baikalox-S30CR [make Baikowski Chimie, France]). As known e.g. from EP-756586B1 (Krell et al.) a high degree of dispersion is obtained by the combined use of several methods, here by 1 h of stirring with simultaneous ultrasonification and subsequent vibration milling for 20 h in a closed polyamide container under a reduced pressure of 60 mbar. On milling, a content of 0.2 wt.-% ZrO_2 was introduced by wear of the used commercial ZrO_2 beads.

Reference samples without ZrO_2 were prepared similarly by 1 h of stirring with simultaneous ultrasonification and subsequent attrition milling for 4 h with 99.99 %

pure submicronstructured Al_2O_3 beads (product of IKTS Dresden) using a container and milling discs of polyethylene.

0.04 wt-‰ of $(NH_4)_2S_2O_8$ (concentration related to Al_2O_3) were added as an initiator after milling. After casting flat discs in glass moulds, final de-gassing of the slurries was achieved at reduced pressure of 200 mbar (air) for 3 h. For consolidation of the slurries, the temperature was then raised to 60 °C to start polymerisation (promoted by the added initiator). This procedure is close to the approach known from EP-756586B1 (Krell et al.).

The wet cast bodies were dried in ambient air for 2 days and then annealed at 800 °C in air to remove the organic additives. On sintering for 2 h in air at 1340 °C the relative density of the ZrO_2-doped samples was increased to 97 %, whereas without ZrO_2 a lower temperature of 1290 °C was sufficient to obtain a closed porosity at a density of 96 %. A final density of > 99.9 % was achieved by hot isostatic pressing (HIP) in argon at 1300 °C / 12 hours for the ZrO_2-doped material. Without ZrO_2 doping the HIP conditions were 1200 °C / 12 h. The average crystal sizes of the transparent corundum microstructures are given by Tab. IV, showing optical data of MgO-doped Al_2O_3 samples (0.03 % MgO), some of them co-doped with zirconia, after HIP and after annealing. After annealing at 1350 °C, only the microstructures *without* ZrO_2 exhibit some larger crystals > 1.3 µm. The particle sizes of most of the ZrO_2 crystals after HIP range between 30 and 100 nm.

After HIP, the diameter of the discs was 28 mm at a thickness of 3 mm. These discs were ground on both parallel sides, first with a diamond wheel with 91 µm grain size, then with a finer diamond grain size of 46 µm. To reduce further the surface roughness, the discs were lapped and polished using successively finer diamond grains of 9, 6 and finally 3 µm. The final thickness of the discs was 0.8 mm.

The real in-line transmission (RIT) and total forward transmission (TFT) were determined with red light (645 nm) with a spectrophotometer type: LCRT-2006-06R/T; make Gigahertz-Optik Corp.. An immersing liquid was used to minimise the influence of surface roughness. The real in-line transmittance was recorded over an angular aperture of 0.5 ° from a straight-line axis. The accuracy of the results for RIT and TFT is in the range of ± 1 % (absolute % units of the transmission data)

Table IV compares the RIT and TFT results of ZrO_2-doped samples according to the present disclosed example 2 with a ZrO_2-free reference: as an example, the table shows that a ZrO_2 concentration of 0.2 % significantly retards the decrease of RIT on high temperature annealing.

In contrast to RIT, the results from Table IV shows there is little effect of annealing on TFT with as without ZrO_2 doping.

TABLE IV.

	as sintered (after HIP)		annealed 1300 °C / 10 h		Annealed 1300 °C / 10 h + 1350 °C / 10h	
	0 % ZrO_2	+ 0.2 % ZrO_2	0 % ZrO_2	+ 0.2 % ZrO_2	0 % ZrO_2	+ 0.2 % ZrO_2
Average grain size	0.62 μm	0.50 μm	not determ.	0.51 μm	1.14 μm	0.67 μm
RIT	51.1 %	64.4 %	41.0 %	61.2 %	30.6 %	42.9 %
TFT	82.6 %	80.6 %	81.3 %	82.6 %	80.4 %	76.5 %

The hardness HV10 of the samples doped with ZrO_2 was after HIP 19.6 GPa, their strength in 4-point bending was 638 ± 51 MPa.

Example 3

A discharge tube was made by slip casting of a slurry prepared according to the process as described in example 1, with 0.04 wt-% ZrO_2. The thus formed shaped body was sintered at a sinter temperature of 1300 ^{0}C during 2 hours, after which it was given an HIP treatment for 2 hours at a temperature of 1200 ^{0}C.

The thus formed discharge tube has a ceramic wall with a average crystal size of 0.7 μm. The ceramic wall material showed a 4 point bending strength of 590 Mpa and had a value for the RIT of 60 %. The total light transmittance of the discharge tube turned out to be 95 %, which indicates that the doped transparent alumina does not absorb light in the visible region of the spectrum.

From the thus formed discharge tube a lamp was made. An example of a discharge lamp having a discharge tube made of alumina according to the invention is described with reference to a drawing. The drawing shows a lamp 10 with discharge tube 1 having a ceramic wall 2 of transparent ceramic according to the invention. The lamp is provided with a partly broken away outer bulb 11. The discharge tube of the lamp is provided with electrodes 60, 70, which are connected by a known in the art leadthrough constructions 6, 7 to current conductors 13, 14. The current conductors are connected on conventional way with electric contacts of a lamp base 12.

In a further experiment an increased HIP temperature of 1400 °C was used to manufacture translucent envelopes with maximum total light transmittance from gelcast samples prepared according to Example 2. The average crystal size of these samples was 0.7 µm after 30 min of isothermal HIP and 1.5 µm after 5 h. The wall thickness of these samples was 1 mm, and there was no polish neither on the outer nor on the inner side of the envelopes. After 5 h HIP, the total light transmittance was 95 % and the average bending strength 590 MPa (4-point bending of bar samples). The stability of the total light transmittance on annealing comes close to that of the TFT data in Table IV.

CLAIMS:

1. Polycrystalline alumina components with an additive of at least 0.001 wt-% ZrO_2 and optionally containing MgO with a concentration of at most 0.3 wt-% characterized in that the alumina has at most 0.5 wt-% ZrO_2 as additive and has an average crystal size ≤ 2 µm, and has a relative density higher than 99.95 % with a real in-line transmission RIT ≥ 30 % measured over an angular aperture of at most 0.5^0 at a sample thickness of 0.8 mm and with a monochromatic wave length of light λ.

2. Polycrystalline alumina components according to claim 1, characterised in that the average crystal size is ≤ 1 µm with a real in-line transmission RIT at least 40 %.

3. Polycrystalline alumina components according to claim 1 or 2, characterised in that the ZrO_2 additive has a concentration between 0.1 wt-% to 0.3 wt-%, boundaries inclusive.

4. Discharge lamp characterized in that the lamp is provided with a discharge tube having a wall of ceramic as claimed in any of the preceding claims.

5. Lamp according to claim 4 characterized in that the discharge tube has an ionisable filling containing a metal halide.

6. Method to form a polycrystalline alumina component as claimed in one of the preceding claims characterized in that the process includes the steps of

- preparing a slurry of corundum power with a mean grain size ≤ 0.2 µm,
- addition of a dopant, selected from zirconia and a zirconium containing precursor,
- casting the slurry in a mould,
- drying and sintering of the thus form shaped body, and
- performing a HIP treatment at a temperature of at least 1150 ^{0}C for at least 2 hours.

7. Method according to claim 6, wherein the dopant is added as finely grained ZrO_2.

8. Method according to claim 6 or 7, wherein the finely grained ZrO_2 dopant has an average particle size of at most 100 nm.

9. Method according to claim 6, 7 or 8 wherein the prepared slurry after the addition of the zirconia dopant is slip cast in a mould.

10. Method according to claim 6, 7 or 8 wherein the prepared slurry after the addition of the zirconia dopant is gel cast in a mould.

ABSTRACT:

The present invention relates to highly dense translucent and transparent aluminium oxide components for applications where, e.g. in the lightening industry, a fine crystal size has to be obtained and stabilised for use at temperatures of 800 °C or more. Disclosed are high-strength polycrystalline alumina products which associate 0.001 – 0.5 weight-% ZrO_2 stabilising a fine crystal size < 2 µm or, preferentially, < 1 µm on use at temperatures of 800 °C or more. The microstructure exhibits an extremely high relative density enabling a high real in-line transmission > 30 % measured over an angular aperture of at most 0.5^0 at a sample thickness of 0.8 mm and with a monochromatic wave length of light λ, preferably of 645 nm.

Fig. 1

1/1